PATENT
Docket No. 361752000500

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the application of:

Keunsuk P. CHANG et al.

Serial No.: 09/715,013

Filing Date: Nov. 20, 2000

For: BIAXIALLY ORIENTED POLYPROPYLENE METALLIZED FILM FOR PACKAGING

Examiner: Kimberly T. Nguyen

Group Art Unit: 1774

SUPPLEMENTAL DECLARATION OF KEUNSUK P. CHANG

Commissioner for Patents
Washington, D.C. 20231

Sir:

Keunsuk P. Chang declares under penalty of perjury under the laws of the United States of America as follows:

1. I am the same Keunsuk P. Chang who submitted a declaration dated April 30, 2002. I refer to that declaration for a statement of my experience and qualifications.

2. I have reviewed the Office Action of November 19, 2002, and have become familiar with the invention and disclosure of U.S. Patent 5,137,955 (Tsuchiya) with the help of Mr. Ken Kurokawa, a colleague here at Toray Plastics (America). Mr. Kurokawa was also a colleague of Mr. Tsuchiya and, in fact, was responsible for helping to commercialize Tsuchiya's films here in the United States in the Rhode Island facility of Toray Plastics (America), Inc. In late December 2002, I discussed the Office Action with Mr. Kurokawa, who then described to me his direct experience with the Tsuchiya film and was able to direct me towards archived files relating to the production results of Tsuchiya's film here in the United States for commercial applications. I

reviewed the documents attached to Mr. Kurokawa's declaration and discussed them with him before preparing this declaration. The purpose of this declaration is to explain the nature of the Tsuchiya film documents found by Mr. Kurokawa.

3. Tsuchiya's film design as disclosed in U.S. Patent No. 5,137,955 requires the use of an additive slip agent of an hydroxy-fatty acid glyceride, preferably glyceryl tri-12-hydroxystearate. The use of this glyceryl tri-12-hydroxystearate ("TG" for short) is essential in Tsuchiya's design in affording good adaptability to vacuum deposition processing such as vapor deposition of aluminum in typical vacuum metallizing processes. Those films which did not contain TG – as shown in Table 1 of Tsuchiya's patent – showed poor adaptability to vacuum deposition, and thus poor metallizing due to metal deposition defects, pinholes, wrinkles, blocking, etc.

4. In the U.S., Toray Plastics (America) tried to commercialize Tsuchiya's film during 1990–1992. The internal designation for Tsuchiya's film was YS-94(W) (a/k/a Y94, Y94W, YS94 for short). YS-94(W)'s composition is shown in attached Exhibit 1, which is a document found in the Torayfan technical library I received from Mr. Kurokawa. It was a 2-layer film, the base or core layer comprising a propylene homopolymer called Exxon PD4292 with 0.02% of an inorganic spherical antiblock of 2 μm diameter called Tospearl 120. The skin layer was comprised of an ethylene-butylene-propylene terpolymer called WF-345 which contained an inorganic spherical antiblock of 4.5 μm size called Tospearl 145, an inorganic antiblock of Syloid silica, oleamide slip agent, and the TG slip agent at 1.5% by weight of this layer. When this film was metallized in 1990, its oxygen and moisture vapor transmission rate values (O2TR and MVTR) were uniformly poor. Exhibit 2 illustrates some of the O2TR and MVTR values reported at that time: Over 2000 cc/m^2/day O2TR and over 6 g/m^2/day MVTR. In comparison with these barrier values, the films of this application exhibit O2TR and MVTR of about 20 cc/m^2/day or less (and typically about 10 cc/m^2 day) and 0.20 g/m^2/day or less respectively. These values are 2 orders of magnitude better in oxygen transmission and 30 times better in moisture transmission than Tsuchiya's film. In terms of barrier durability, Tsuchiya's film would not have exhibited any meaningful barrier durability values as its flat sheet or unelongated

sheet barrier was already so high that it rendered any measurements of barrier durability moot. The film of this application has a barrier durability at 9% elongation of less than 46.5 cc/m^2/day (and typically less than 30 cc/m^2/day) which is still at least 40 times better than Tsuchiya's film in an unelongated state.

5. By 1992, Tsuchiya's film design did not improve significantly in barrier properties. Exhibit 3 indicates this clearly. The YS-94 product still had reported O2TR of 340-440 cc/m^2/day and MVTR of over 2.0 g/m^2/day. Even over a range of optical densities from less than 1.0 to as high as almost 4.0, both the oxygen and moisture barrier properties of Tsuchiya's film remained poor. Even though Tsuchiya's patent cites an optical density range of 0.5-5.0, all his examples are metallized at a 2.0 optical density, less than the range specified by this application. However, even when Tsuchiya's film was metallized at optical densities approaching 4.0 as shown in Exhibit 3, barrier and barrier durability properties remained very poor. The film of this application, in contrast, continued to be at least a magnitude better in oxygen and moisture barrier, whether considered as a flat sheet or elongated under stress to demonstrate superior barrier durability.

6. The film of this application does not contain any TG or any other migratory slip additives as Tsuchiya's film does. In fact, Comparative Example 10 in Table 1 of U.S. Patent No. 5,137,955 is closest in design to Chang's film except for optical density target, yet Tsuchiya reports that such a film was "poor" for adaptability to vacuum deposition. We have not observed similar shortcomings in the development of the film of this application. This film has proven itself to be readily adaptable to vacuum deposition; its outstanding barrier properties and sales volume of over 1 million pounds in 2002 attest to its commercial success. In my opinion as a person experienced in this field, it is possible that the inclusion of TG in Tsuchiya's film as a migratory organic fatty acid additive had a detrimental effect on barrier properties in that it would have migrated or transferred easily to the metallizing surface. Upon metallization, the TG – being of low melting point and low molecular weight -- could flash evaporate under the heat of the condensing aluminum vapor and essentially blow holes through the metal-deposited layer, thus compromising barrier properties. Thus, Tsuchiya's film would intrinsically be unable to

demonstrate good barrier or good barrier durability properties. Moreover, Tsuchiya's patent clearly indicates that the best performance for "adaptability to vacuum deposition" must be films that include TG; without TG, according to Tsuchiya, such adaptability would be poor and unviable for commercialization. Thus, persons of ordinary skill in this art – based on Tsuchiya's own data and that of Toray's own commercialization efforts at the time – would not have been motivated at the time to modify Tsuchiya's base film design and optical density parameters to provide improvements in barrier durability. Furthermore, as indicated by Toray's internal data on commercializing Tsuchiya's film design, Tsuchiya's film intrinsically did not contain the high barrier or barrier durability properties of the film of this application.

7. Indeed, as explained in my first declaration dated April 30, 2002, the industry as a whole did not recognize the improvements that high optical density on the proper basefilm design could deliver as shown by AIMCAL proceedings cited in the declaration. Thus, the current invention's improvement in barrier and barrier durability was unexpected and unanticipated from Tsuchiya and the other prior art.

8. In reviewing prior art regarding basefilm design for metallized films, I have come across two references of interest. One is U.S. Patent No. 4,345,005 by All et al., issued August 17, 1982. All states in Column 1, line 30, that "...avoidance of the use of slip agent in the polypropylene film acts to provide enhanced adhesion of such film to a metallized coating." However, All refers only to metal adhesion improvement, not barrier properties or barrier durability. Nor does he refer to any optical density target or range for metal deposition. Thus, All would not have provided motivation to develop a barrier or barrier durable film with the claimed combination of metallization optical density and film design. In the absence of any optical density specification, All's film would not have intrinsically been barrier durable.

9. The second reference is U.S. Patent No. 6,033,786 by Fatica et al., issued March 7, 2000. Fatica states in Column 6 lines 43-51 that "... it is advisable to use little or none of the additives such as slip agents or antistats ... [which] can interfere with establishing an effective metal coating for enhancing the oxygen and moisture barrier properties of the film." However, Fatica's films are all metallized at an optical density range of 2.0 to 2.3, well below the specific

optical density of 2.6 minimum of the present invention. Thus, Fatica's films would not have been anticipated to demonstrate the improved barrier and barrier durability properties of the present invention, nor would Fatica have been motivated to provide high optical density. Again, I cite my previous declaration where I explain that the prevailing notion at the time was that higher optical densities provided negligible or even detrimental properties.

10. The third reference, Comer U.S. Patent No. 6,139,930, claims a very smooth "barrier receiving surface" of an RMS roughness of at least 12 nm with standard deviation of 1.2 and discloses that the RMS roughness of this surface is preferably between 14 and 15 nm (column 9, lines 18-20) and the Ra of this surface is at least about 9 nm and is preferably between 10 to 12.5 nm (column 9, lines 25-28). The film disclosed in this application typically shows an RMS roughness of 60-70 nm and Ra of 30-50nm. Therefore, our film is significantly rougher than Comer's film and yet shows equivalent barrier properties in its unelongated form.

11. Comer does not disclose or claim an optical density or metal thickness range. In the specification and examples, however, Comer specifies an aluminum thickness of 75-100 Å, preferably 100-350 Å (column 8, line 67 – col. 9, line2). In Comer's examples, the Al thickness is shown to be about 135 Å (column 12, line 38). The thickness of the aluminum film of this application is typically 500 Å or more as described in our specification, preferably 600-700 Å. Therefore, our metallized film has a significantly thicker aluminum layer (claimed in terms of optical density) that is one of the key features that gives superior barrier AND barrier durability via elongation. Comer's film is much thinner aluminum thickness and does not claim or mention barrier elongation durability (although Comer cites improvement in Gelbo flex barrier improvement -- which is not the same as elongated barrier -- in column 18, Table 7). Nevertheless, Comer's reported oxygen transmission value of 300cc/m^2/day after flexing is about a magnitude higher than our elongated barrier values.) Therefore, Comer's film is not likely to show the barrier durability that our film does.

12. Comer does mention (as does Fatica) that using migratory slip additives can be detrimental to barrier properties (column 7, lines 42 - 51). However, as with Fatica, the

thickness of Comer's aluminum layer (and by extension, optical density) is less than our film's and, thus, Comer's would not have been expected to show barrier durability properties of the level claimed in this application.

13. Finally, Comer discloses corona or flame treatment as oxidative treatment methods. Column 8, lines 29-54. As a result, no nitrogen functional groups would be expected to occur at the treated surface of Comer's film, in contrast with the film claimed in this application.

14. Attached Exhibits 1-3 are those that Mr. Barry Bretschneider and I discussed with Examiners Nguyen and Kelly at the interview of January 21, 2003.

I declare under penalty of perjury under the laws of the United States that the foregoing is true and correct. Executed at North Kingstown, Rhode Island this 24 day of February, 2003.

Keunsuk P. Chang

W：ベースのみ海外原料
Z：ベース，ラミ両方とも海外原料

5．TREAの原料組成

フィルム構成	YS－74（W）	YS－74（Z）	YS－94（W）	YS－94（Z）
ベース層	・無添加ホモ EXXON-PD-4292 80% ・ゼオライトマスター 20% 三東圧を使用したいが米国事情によりサイロイドをベースとする。但しヘイズ基準であるゆえにSA剤を直投し易滑性をコントロールする ・ステアリン酸アミド Humko-Kemamide S 直投 0.1%	同左	・無添加ホモ EXXON-PD-4292 90% ・ゼオライトマスター 10% 三東圧・・・以後同左 トスパールのFDA が認可された時点で、トスパール－120の 0.5%マスターをEXXON に作らせ、TREAで 0.02%に希釈する。 ゼオライト 0.03%＝トスパール 0.02%	・無添加ホモ EXXON-PD-4292 100% ・トスパール－120 0.02%
ラミ層	・シリカ・アミド入りコポリマー 住化WF-345A 100% サイロイド 0.35% OA 0.25% エージング時間の短縮による蒸着ブロッキング防止にはOA+SA (SA0.1%)トータル有機易滑剤は0.25%となる。	・シリカ・アミド入りコポリマー FINA-Z-9470A 100% サイロイド 244 0.35% OA=Humko Kemamide U 0.25% 同左	・シリカ入りコポリマー 住化WF-345改 100% （サイロイド 0.35%） ・トスパール-145 0.25% ・TG 1.5% ・OA 0.1% これら添加剤を住化で混合。	・シリカ，トスパール，TG，OA入りコポリマー FINA-Z-9470B 100% サイロイド 0.35% トスパール-145 0.25% TG 1.5% OA 0.1% FINAに全添加したものを作らせる。
備考	・Eフレーク，再ペレはベース層へ回収 ・ゼオライトマスターは，EXXON が対応できる時期から水沢化学より購入	同左	・このケースは，FINAがYS-94(Z)用のラミ原料を作れない場合である。作れる場合はYS-94(Z)で進める。	・YS-94(Z)をスタートする時は，トスパールのFDA 認可と特許のライセンス受けを実現させておく。

EXHIBIT
Serial No. 09/715,013
1

9-11-1990

TO TPA
Mr. SUZUI

From Toray Shiga
Film Tech. Dept.
Mr. KOEBISU
稚代
村
2.9.11
上

Frito-Lay Vend Pack Test 60G Y94W
MVTR 異常品の解析について

現時点での評価分析状況は以下の通りです。

1. MVTR NG, OK品の原反フィルム特性（図1～3）
両者に有意差なし

2. MVTR NG, OK品の蒸着フィルムの比較
（図4, 5）
(1) 蒸着フィルム単体のMVTR測定で有意差あり
(2) O_2TRについても有意差あり
(3) NaOH溶解性，耐スクラッチ性については Mr. SUZUI とのMeeting（日本）時の資料の通りです。
(4) ドライラミ強度
濡れ張力の違いによるものと推定
(5) AL層の構造
9/12 9:00 amより TRC評価分析状況について打合せを予定しています。

3. NaOH溶解性とMVTRの定量的関係
NaOH溶液の濃度変更、表面有機物洗浄後のサンプルについて等、調べていますが明確な傾向がでていません。

4. Printpackラミ加工品、ドライラミ品のMVTR測定（MOCON法）（図6）
TPAと下地で対応とれているが、下地データの方が平均して 0.005 g/100 in²·24hr 高目に出ている（100°F × 90% RH）

以上

図1

MVTR異常品の解析
(1) フィルム特性

項目		単位	方向,面	70G Y74W (ラミ厚:3.5μ)	60G Y94W (ラミ厚:1.5μ) MVTR NG品	60G Y94W (ラミ厚:1.5μ) MVTR OK品	70G Y94W (ラミ厚:1.5μ)	70G HST622 (ラミ厚:1.0μ)
フィルム中のアミドの定量		%	—	OA 0.019 SA 0.062 EA 0.010以下	(TG−12 1.5)	→	→	OA 0.006以下 SA 0.004以下 EA 0.026以下
フィルム硬さ		—	—	別添写真参照	有意差なし		別添写真参照	別添写真参照
ボイド観察		—	—	サイズ大:4μ以上~23μ 1800個/mm²	サイズ大:4μ以上~41μ 190個/mm²	サイズ大:4μ以上~41μ 190個/mm²	サイズ大:4μ以上~35μ 230個/mm²	サイズ小:4μ以上~ 460個/mm²
MVTR	原反 蒸着品	g/m²·24hr	MOCON	6.60 —	7.02 2.84	6.99 1.09	6.84 —	6.95 —
O_2TR	(73°F.0%RH)	cc/m²·24hr		—	2010	2010	—	—
屈折率		—	nMD nTD nZD $\bar{n}$ Δn	1.5031 1.5188 1.4971 1.5063 0.0157	1.5021 1.5171 1.4970 1.5054 0.0150	1.5023 1.5170 1.4971 1.5055 0.0147	1.5029 1.5180 1.4992 1.5061 0.0163	1.5023 1.5191 1.5012 1.5075 0.0168
3次元表面粗さ	Rma Ra Rz	nm	A面	2475 32.0 1740	925 27.0 680	550 25.0 450	2450 29.7 1275	1413 29.8 680
	Rma Ra Rz	nm	B面	875 43.2 670	2038 68.8 1993	2163 59.0 2000	2038 51.3 1518	888 37.5 725
TMA		℃	MD TD	118.4 128.3	116.3 126.9	117.0 124.5	118.0 124.2	117.3 106.7
熱収縮率	100℃×15分	%	MD/TD	1.6/−0.2	1.8/−0.1	1.7/−0.1	1.6/−0.1	1.9/0.7
	120℃×15分	%	MD/TD	3.3/0.6	3.4/0.8	3.2/0.7	3.2/0.8	3.6/3.0
	140℃×15分	%	MD/TD	7.0/5.1	6.8/5.7	6.4/5.6	6.4/5.4	7.6/11.1

*無機微粒子の分析

・Y94W ベース層:真球シリカ2μφ 200ppm
ラミ層:真球シリカ4μφ 3000ppm
サイロイド244 3500ppm

・Hercules HST622 3層トータル
Mg:104ppm Ti: 1ppm
Al: 90ppm Na:数10ppm

** A面:処理面、B面:非処理面

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図2

(1) Film Properties

Properties		Unit	Direction, side	70G Y74W (sealant layer :3.5μ)	60G Y94W (sealant layer :1.5μ) Lot no.01300419 (NG)	60G Y94W (sealant layer :1.5μ) Lot no.02300419 (OK)	70G Y94W (sealant layer :1.5μ)	70G HST62[illegible] (sealant layer :1.[illegible]
amount of amide		%	—	OA 0.019 SA 0.062 EA <0.010	(TG−12 1.5)	→	→	OA <0.006 SA <0.004 EA <0.026
film hardness		—	—	attached	no significant difference		attached	attached
void	size density	μ /mm²	—	large :4~23 1800	large :4~41 190	large :4~41 190	large :4~35 230	small :4~17 460
MVTR	clear film metalized film	g/100in²·24hr	MOCON	0.426 —	0.453 0.183	0.451 0.070	0.441 —	0.448 —
O_2TR	(73°F.0%RH)	cc/100in²·24hr		—	130	130	—	—
refractive index		—	nMD nTD nZD $\bar{n}$ Δn	1.5031 1.5188 1.4971 1.5063 0.0157	1.5021 1.5171 1.4970 1.5054 0.0150	1.5023 1.5170 1.4971 1.5055 0.0147	1.5029 1.5180 1.4992 1.5061 0.0163	1.5023 1.5191 1.5012 1.5075 0.0168
surface roughness	Rms Ra Rz	nm	A	2475 32.0 1740	925 27.0 680	550 25.0 450	2450 29.7 1275	1413 29.[illegible] 680
	Rms Ra Rz	nm	B	875 43.2 670	2038 68.8 1993	2163 59.0 2000	2038 51.3 1518	888 [illegible] 72[illegible]
TMA		°F	MD TD	245 283	241 260	243 256	244 256	243 224
heat shrinkage	212°F×15min	%	MD/TD	1.6/−0.2	1.8/−0.1	1.7/−0.1	1.6/−0.1	1.9/0.[illegible]
	248°F×15min	%	MD/TD	3.3/0.6	3.4/0.8	3.2/0.7	3.2/0.8	3.6/3.[illegible]
	284°F×15min	%	MD/TD	7.0/5.1	6.8/5.7	6.4/5.6	6.4/5.4	7.6/11.[illegible]

* Analysis of inorganic particles

· Y94W Base layer :spherical silica(2μφ) 200ppm
Sealant layer:spherical silica(4μφ) 3000ppm
syloid244 3500ppm

· Hercules HST622 Mg:104ppm Ti: 1ppm
Al: 90ppm Na:10~50ppm

** A : treated side, B : non-treated side
MD :machine direction, TD : transverse direction

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図 3

(1) Film Properties

Properties		Unit	Direction, side	70G Y74W	60G Y94W Lot no.01300419 (NG)	60G Y94W Lot no.02300419 (OK)	70G Y94W	70G HST62
film hardness		–	–	attached	no significant difference		attached	attached
void	size density	μ /mm²	–	large : 4～23 1800	large : 4～41 190	large : 4～41 190	large : 4～35 230	small : 4～17 460
MVTR	clear film metalized film	g/100in²·24hr	MOCON	0. 426 –	0. 453 0. 183	0. 451 0. 070	0. 441 –	0. 4[illegible]8
O₂TR	(73°F, 0%RH)	cc/100in²·24hr		–	130	130	–	–
refractive index		–	nMD nTD nZD n̄ Δn	1. 5031 1. 5188 1. 4971 1. 5063 0. 0157	1. 5021 1. 5171 1. 4970 1. 5054 0. 0150	1. 5023 1. 5170 1. 4971 1. 5055 0. 0147	1. 5029 1. 5180 1. 4992 1. 5061 0. 0163	1. 5023 1. 5191 1. 5012 1. 5075 0. 0168
surface roughness	Rma Ra Rz	nm	A	2475 32. 0 1740	925 27. 0 680	550 25. 0 450	2450 29. 7 1275	1413 29. 680
	Rma Ra Rz	nm	B	875 43. 2 670	2038 68. 8 1993	2163 59. 0 2000	2038 51. 3 1518	888 37. 725
TMA		°F	MD TD	245 263	241 260	243 256	244 256	24[illegible] 224
heat shrinkage	212°F×15min	%	MD/TD	1. 6/−0. 2	1. 8/−0. 1	1. 7/−0. 1	1. 6/−0. 1	1. 9/0.
	248°F×15min	%	MD/TD	3. 3/0. 6	3. 4/0. 8	3. 2/0. 7	3. 2/0. 8	3. 6/3.
	284°F×15min	%	MD/TD	7. 0/6. 1	6. 8/5. 7	6. 4/5. 6	6. 4/5. 4	7. 6/11.

* A : treated side. B : non-treated side
** MD : machine direction. TD : transverse direction

Mr. Takashima → Mr. Shiyama → Kan

（保）

㊙

コピー禁

大畏

July 20 '92

Ｆ研ノート　第92－3号

平成　４年　４月１７日

（配布先）

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松田　　薫

表題：アルミ蒸着“トレファン”のバリア性改良　（No.１）

（ガスバリア性に及ぼす蒸着条件因子の把握）

（要旨）

“トレファン”タイプ３水準、蒸着装置２種類を使用し、蒸着因子面からのバリア性向上について検討した。

（１）使用原反により大きくバリア性が異なり、Ｙ７４６では安定したバリア性が得らたものの、ＹＳ９４はＣＥ処理の裏移りのためか、蒸着ムラが発生し、ピンホールも多発して、バリア性は非常に悪いものであった。（添加剤のブリードアウトなどの悪要因が考えられる。）

（２）ｍ／ｃ、蒸着圧力、基板温度、蒸着速度の要因の中で、基板温度がバリア性に最も大きく影響し、ＰＰのＴｇ（－１０℃）以下の基板温度では十分なバリア性（MVTR）は発現しないが、基板温度を上げることによりバリア性は向上する。

蒸着装置、蒸着圧力、蒸着速度のバリア性に及ぼす影響は小さかった。

（３）今回の検討においては、前グロー処理はマイナス要因となり、バリア性（ＭＶＴＲ）向上には至らなかった。

発行	
研究員	主研
永井	今井

EXHIBIT

Serial No. 09/715,013

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表1　"トレファン"ベース　Al蒸着膜のRs、Tr、OD値、WVTR

「基板温度の影響：T20213シリーズ」

m／c：　EV－1　　原反　："トレファン"YS94#20　LOT.12263047002

ルツボ：アルミナ2元　　アルミ：純度　4N（真空冶金製）　　加速電圧：17.5kv

SampleNo.	蒸着条件 基板温度 (℃)	圧力(Torr) 上室	圧力(Torr) 下室	電流 (A)	速度 (m/min)	Al付着量 (μg/cm²)	Al膜厚[*1] 理論 (Å)	表面電気抵抗値 Rs (Ω/□)	光線透過率 λ=400nm (%)	光線透過率 λ=550nm (%)	OD値[*2] —	換算 透過率 (%)	WVTR 袋法 (g/m²day)	WVTR 山武法	O₂TR CC/m²day
			x10⁻⁴												
T20213-1	-20	1.7	0.85	0.33	4			3x10⁰	0	0	2.84	0.14	2.60		
2		1.7	0.85		6	11.3	420 (550) TEM	4	0.3	0.2	2.43	0.37	2.53	2.4	440
3		1.6	0.75		8			6	1.5	0.9	1.87	1.35	2.54		
4		1.5	0.7		10			7	3	1.6	1.49	3.24	2.56		
5		1.4	0.7		12			8	5.5	2	1.22	6.03	2.59		
6		1.4	0.7		16			1x10¹	13.5	8	0.90	12.6	2.71		
7		1.3	0.7		20			2	22	14	0.69	20.4	2.73		
8		1.3	0.7		2			2x10⁰	0	0	3.57	0.03	3.26		
9	15	1.4	0.6	0.33	2			2	0	0	3.94	0.01	2.98		
10		1.3	0.5		4			3	0	0	2.99	0.10	2.56	2.7	
11		1.2	0.5		8			7	2.5	1.4	1.51	3.09	2.56		
12		1.2	0.5		12			1x10¹	12.5	7.5	0.90	12.6	2.60		
13		1.2	0.5		16			2	24	15	0.63	23.4	2.79		
14		1.1	0.5		20			2	35.5	24	0.48	33.1	2.82		
15	45	1.2	0.45	0.33	2			2x10⁰	0	0	3.99	0.01	2.22		
16		1.2	0.45		4	11.3	420 (550) TEM	4	0.2	0.1	2.55	0.28	2.22	2.1	340
17		1.2	0.45		8			7	6	3.3	1.23	5.89	2.47		
18		1.1	0.45		12			1x10¹	19	11.5	0.74	18.2	2.52		
19		1.0	0.4		16			2	33.5	22	0.54	28.8	2.68		
20		1.0	0.4		20			3	46	33	0.35	44.7	3.00		
VS94 #20													4.65		

表2　"トレファン" ベース　Al蒸着膜のRs、Tr、OD値、WVTR

「グロー処理の効果：T20214シリーズ」

m／c：　EV－1　　原反　："トレファン" YS94＃20　LOT. 12263047002

ルツボ：アルミナ2元　　アルミ：純度　4N（真空冶金製）　　加速電圧：17．5kv

SampleNo.	グロー条件 ガス流量 (l/分)	グロー条件 圧力(Torr) 上室	グロー条件 圧力(Torr) 下室	グロー条件 電流 (A)	グロー条件 電圧 (V)	蒸着条件 電流 (A)	蒸着条件 速度 (m/min)	表面電気抵抗値 Rs (Ω/□)	光線透過率 λ=400nm (%)	光線透過率 λ=550nm (%)	OD値 －	換算透過率 (%)	WVTR 袋法 (g/m²day)	WVTR 山武法
	Ar	10^{-3}	10^{-4}											
T20214-1	0.08	4.0	5.5	0.1	450	0.4	2	3×10^{0}	0	0	3.33	0.05	3.67	
2	〃						4	2	0	0	3.10	0.08	2.97	
3	〃						8	4	0.85	0.4	1.99	1.02	2.28	2.9
4	〃						12	6	4.3	2.3	1.34	4.57	2.27	
5	〃						16	1×10^{1}	12	7.5	0.96	11.0	2.33	
6	0.14	6.0	8.0	0.2	600	0.4	2	2×10^{0}	0	0	3.10	0.08	4.16	
7	〃						4	4	0.2	0.1	2.45	0.35	3.13	2.8
8	〃						8	7	4.5	2.3	1.33	4.68	3.25	
9	〃						12	1×10^{1}	14	8.5	0.85	14.1	2.29	
10	〃						16	2	25	16	0.62	24.0	2.36	
11	0.14	6.0	8.0	-	-	0.4	2	3×10^{0}	0	0	3.10	0.08	4.13	
12	〃						4	5	0.75	0.55	2.03	0.93	3.26	4.3
13	〃						8	9	8.8	5	1.03	9.33	2.69	
14	〃						12	1×10^{1}	18.5	12	0.74	18.2	2.68	
15	〃						16	2	30.5	20	0.54	28.81	2.82	
16	-	0.7	0.4	-	-	0.4	2	1×10^{0}	0	0	3.72	0.02	3.11	
17	-						4	2	0	0	2.72	0.19	2.66	
18	-						8	5	1.1	0.6	1.89	1.29	2.39	
19	-						12	8	6	3.4	1.22	6.03	2.48	
20	-						16	1×10^{1}	14	8.5	0.87	13.5	2.55	
21	-						20	2	24.5	16	0.65	22.4	2.54	
22	-						2	1×10^{0}	0	0	3.85	0.01	2.96	

表3　"トレファン" ベース　Al蒸着膜のRs、Tr、OD値、WVTR

「高速蒸着、アルミ純度の影響：T20220シリーズ」

m／c：　EV−1　　原反　："トレファン" γS94#20　LOT. 12263047002　基板温度：−25℃

ルツボ：アルミナ2元　　アルミ：①純度　4N（真空冶金製）②Si5%含有　　加速電圧：17.5kv

SampleNo.	蒸着条件 Al純度	圧力(Torr) 上室	圧力(Torr) 下室	電流 (A)	速度 (m/min)	表面抵抗値 Rs (Ω/□)	光線透過率 λ=400nm (%)	光線透過率 λ=550nm (%)	OD値 —	換算透過率 (%)	WVTR 袋法 (g/m²day)
			×10⁻⁴								
T20220-1	4N	1.6	1.0	0.4	10	0.6×10⁰	0	0	3.84	0.01	2.26
2					20	1	0	0	3.62	0.02	1.95
3					30	1.5	0	0	3.41	0.04	2.04
4					40	2	0	0	2.90	0.13	2.02
5					50	2.5	0.3	0.2	2.40	0.40	2.12
6					60	3	0.8	0.5	2.04	0.91	2.15
7					80	4	2.8	1.5	1.55	2.82	2.29
8					70	4	1.4	0.7	1.83	1.48	2.22
9		1.3	0.6	0.2	4	3	0	0	3.08	0.08	2.33
10					8	6.5	2.8	1.6	1.57	2.69	2.42
11		1.1	0.55		6	5	0.6	0.3	2.17	0.68	2.28
12					7	5	0.9	0.5	1.95	1.12	2.37
13					10	8	4.1	2.5	1.41	3.89	2.40
14					12	9	6.7	3.7	1.18	6.61	2.43
15	Si5%	1.3	0.8	0.4	50	3	0.1	0.05	2.59	0.26	2.05
16					80	4	1.7	0.9	1.70	2.00	2.13
17					70	3	0.75	0.4	2.01	0.98	2.18
18					60	3	0.3	0.15	2.35	0.45	2.10
19					40	2	0	0	3.42	0.04	1.91
20		1.3	0.7		20	0.7	0	0	3.96	0.01	1.74
21		1.2	0.6	0.2	8	3	0.15	0.1	2.57	0.27	2.22
22					12	4.5	1.15	0.6	1.86	1.38	2.26
23					16	6	4.3	2.3	1.39	4.07	2.28
24					14	6	1.9	1.1	1.68	2.09	2.26
25		1.0	0.5		18	7	4.8	2.6	1.32	4.79	2.28
26		1.1	0.5		10	3	0.2	0.1	2.44	0.36	2.12

表4. "トレファン"ベース Al蒸着膜のRs、Tr、OD値、WVTR
「EB−X基礎データ：T20227シリーズ」

m／c：EB−X　　原反："トレファン"YS94#20　LOT.12263047002
ルツボ：アルミナ　　アルミ：純度 4N（真空冶金製）　加速電圧：10Kv
到達圧力：上室 1.9×10^{-4}Torr　下室 1.9×10^{-6}Torr（排気時間 4hr）

サンプルNo.	蒸着条件			表面抵抗	光線透過率		OD値	換算	WVTR
	下室圧力	電流	蒸着速度	Rs	Tλ = 400nm	Tλ = 550nm		透過率	袋法
	Torr	A	m/min	Ω／□	%	%	−	%	g/m² day
YS94#20	（×10^{-6}）	−	−	−	−	−	−	−	4.46
T20227-1	3.0	0.15	2	2	0	0	3.77	0.02	2.58
2	〃	〃	4	5	2.15	1.2	2.09	0.81	3.02
3	2.5	〃	8	9	10.75	6	0.97	10.72	3.33
4	〃	〃	6	4	1.5	0.8	1.76	1.74	3.08
5	2.1	0.10	2	3×10	47	32.5	0.35	44.67	3.50
6	〃	〃	1	−	−	−	−	−	−
7	2.2	0.20	4	3	3	0.15	3.23	0.06	2.76
8	3.2	〃	8	1	0	0	3.88	0.01	2.26
9	2.8	〃	12	3	0.25	0.2	2.43	0.37	2.55
10	3.0	〃	16	2×10	27	16.5	0.64	22.91	3.27
11	2.2	0.125	2	2×10	18	10.5	0.76	17.38	3.32
12	〃	〃	1.5	1×10	12	6.75	0.85	14.13	3.15
13	1.9	〃	1	1×10	14.5	8.25	0.83	14.79	3.25
14	〃	0.175	2	2	0	0	3.29	0.05	2.68
15	〃	〃	4	6	3.8	2.05	1.45	3.55	3.02
16	〃	〃	8	1×10	21.25	12.5	0.66	21.88	3.31
17	2.0	〃	6	1×10	12.75	7.25	0.86	13.80	3.24
18	〃	0.15	4	2×10	31	17	0.49	32.36	3.35
19	2.1	0.20	4	3	0	0	2.70	0.20	2.81
20	2.0	〃	8	1×10	31	7.5	0.90	12.59	3.21
21	〃	〃	6	9	10	5.5	0.95	11.22	3.16
22	1.9	〃	4	6	6	3.3	1.18	6.61	3.27
23	〃	〃	2	4	1.15	0.6	1.81	1.55	3.02

表5　"トレファン"ベース　Al蒸着膜のRs、Tr、OD値、WVTR
「EB－X基礎データ：T20310シリーズ」

m／c：EB－X　　原反　："トレファン"YS94#20　LOT.12263047002
ルツボ：アルミナ　　アルミ：純度　4N（真空冶金製）
到達圧力：上室　5×10⁻⁴Torr　下室　5×10⁻⁵Torr（排気時間　1hr）

ｻﾝﾌﾟﾙNo.	蒸着条件			表面抵抗	光線透過率		OD値	換算	WVTR
	下室圧力	電流	蒸着速度	Rs	Tλ = 400nm	Tλ = 550nm		透過率	袋法
	Torr	A	m/min	Ω／□	%	%	—	%	g/m² day
YS94#20	(×10⁻⁵)	—	—	—	—	—	—	—	4.40
T20310-1	5.6～6.2	0.25	2	1	0	0	4.21	0.006	2.23
2	5.8～5.5	〃	4	3	0.2	0.1	2.58	0.26	2.70
3	5.2～4.8	〃	6	5	2.8	1.5	1.50	3.16	2.96
4	4.4～4.2	〃	5	7	7.2	3.95	1.31	4.90	2.97
5	4.0	〃	3	5	1.0	0.5	1.88	1.32	2.86
6	3.0	0.35	4	1	0	0	3.95	0.01	2.15
7	3.5	〃	8	2.5	0.3	0.15	2.68	0.21	2.58
8	3.9	〃	12	4	2.3	1.75	1.56	2.75	2.90
9	4.1	〃	16	7	9	5	1.14	7.24	2.97
10	3.7	〃	10	4	2.5	1.35	1.51	3.09	2.88
11	3.8	〃	9	3	1.15	0.6	1.78	1.66	2.77
12	3.4 ↘	0.25	2	10	10.25	5.5	0.91	12.30	3.10
13	2.8 ↘	〃	1.5	7	5.25	2.8	1.23	5.89	3.20
14	2.3 ↘	〃	1.2	6	2.5	1.35	1.68	2.09	2.93
15	2.1 ↘	〃	1	5	1.6	0.9	1.73	1.86	2.79